



**Calhoun: The NPS Institutional Archive**  
**DSpace Repository**

---

Theses and Dissertations

1. Thesis and Dissertation Collection, all items

---

1955-05-16

## The point emitter as a positive-ion source

Herron, Russel Gardner

Monterey, California: U.S. Naval Postgraduate School

---

<http://hdl.handle.net/10945/14503>

---

*Downloaded from NPS Archive: Calhoun*



<http://www.nps.edu/library>

Calhoun is the Naval Postgraduate School's public access digital repository for research materials and institutional publications created by the NPS community. Calhoun is named for Professor of Mathematics Guy K. Calhoun, NPS's first appointed -- and published -- scholarly author.

**Dudley Knox Library / Naval Postgraduate School**  
**411 Dyer Road / 1 University Circle**  
**Monterey, California USA 93943**

THE POINT EMITTER AS A POSITIVE-ION SOURCE

---

RUSSELL GARDNER HERRON

Library  
U. S. Naval Postgraduate School  
Monterey, California





UNIVERSITY OF CALIFORNIA

Radiation Laboratory  
Berkeley, California

Contract No. W-7405-eng-48

THE POINT EMITTER AS A POSITIVE-ION SOURCE

Russell Gardner Herron

(M.S. Thesis)

May 16, 1955

Submitted in partial fulfillment  
of the requirements for the degree of

MASTER OF SCIENCE

in

PHYSICS

United States Naval Postgraduate School  
Monterey, California

UNIVERSITY OF CALIFORNIA  
LIBRARY  
Berkeley, California  
Contract No. W-7404-ENG-28

THE HOUT BATTERY AS A POSITIVE-ION SOURCE  
B. H. HOUT  
(M. S. Thesis)  
May 16, 1952

Submitted in partial fulfillment  
of the requirements for the degree of  
MASTER OF SCIENCE  
in  
PHYSICS  
JAMES EARL HOUT, Ph.D.  
Berkeley, California

THE POINT EMITTER AS A POSITIVE-ION SOURCE

\* \* \* \* \*

Russell G. Herron



THE POINT EMITTER AS A POSITIVE-ION SOURCE

by

Russell Gardner Herron

Lieutenant, United States Navy

Submitted in partial fulfillment  
of the requirements  
for the degree of  
MASTER OF SCIENCE  
IN  
PHYSICS

United States Naval Postgraduate School  
Monterey, California

1 9 5 5

11-13  
H52

THE FIRST EDITION OF A PAPER FOR PAPER

THE FIRST EDITION OF A PAPER FOR PAPER

by

Walter G. (London, 1870)

London, 1870. 1 vol. 8vo. 1/6

Submitted to the Library of the  
of the University of  
for the purpose of  
MASTERS OF ARTS  
IN  
PHYSICS

Printed at the University Press, Cambridge

1871

Library  
U. S. Naval Postgraduate School  
Monterey, California

This work is accepted as fulfilling  
the thesis requirements for the degree of

MASTER OF SCIENCE

IN

PHYSICS

from the

United States Naval Postgraduate School



## ABSTRACT

A needle-shaped tungsten "point" filament has been used as a positive ion source. Hydrogen ions were produced experimentally at room temperature from the point emitter under the effect of high electric fields. The electron field-emission microscope is recommended as a tool to augment data on adsorption, migration, and vaporization characteristics of the heavier elements. The preferential transfer of  $\text{Cm}^{242}$  as ions from an emitter point at  $1600^\circ\text{K}$  was observed. The point emitter is shown to be a source of positive ions for solid samples. Application of point-emitter techniques to mass spectroscopy is considered probable upon solution of the sample-replenishment problem.



## PREFACE

The field-emission electron microscope, which uses high electric fields to achieve electron emission, has frequently been used in the study of the fundamental physics of electron emission. Recently work has been undertaken by various investigators to determine the feasibility of using similar high field techniques with reversed polarity to obtain positive ions. One such program began in the summer of 1954 at the Radiation Laboratory of the University of California at Berkeley. Mr. Fred L. Reynolds of the Chemistry Division formulated the problem and served as project leader. The author served as research assistant to Mr. Reynolds.

It is the aim of this paper to report the progress of this project to date.

The writer wishes to acknowledge and thank Fred L. Reynolds for his cooperative supervision of the project; Maynard C. Michel and George W. Kilian for their valuable help in the preparation of radioactive samples and construction of experimental equipment; and Professor Norman L. Oleson of the U.S. Naval Postgraduate School for his assistance in the preparation of this paper.



## TABLE OF CONTENTS

Item	Title	Page
Abstract	. . . . .	ii
Preface	. . . . .	iii
List of Illustrations	. . . . .	v
List of Tables	. . . . .	v
Chapter I	Introduction . . . . .	1
Chapter II	Emitter Fabrication . . . . .	4
Chapter III	Fabrication of Fluorescent Screen Tubes .	7
Chapter IV	The Vacuum System . . . . .	10
Chapter V	Experimental Studies	
	1. Ionization of hydrogen . . . . .	13
	2. Fluorescent-screen patterns of point emitters . . . . .	16
	3. Transfer of solid samples as ions .	17
Chapter VI	Discussion of Results . . . . .	22
Bibliography	. . . . .	23
Appendix I	Emitter Field Calculations . . . . .	23



## LIST OF ILLUSTRATIONS

Figure		Page
1.	A field emission microscope . . . . .	2
2.	Emitter point: electron micrograph . . . . .	6
3.	Fluorescent-screen tube . . . . .	9
4.	The vacuum system . . . . .	12
5.	Electrometer collection tube . . . . .	14
6.	Solid sample transfer tube . . . . .	19
7.	Field calculation diagram . . . . .	25

## LIST OF TABLES

1.	Ion Transfer Studies ( $\text{Cm}^{242}$ ) . . . . .	20
----	--	----



## CHAPTER I

### INTRODUCTION

The essential features of a field-emission microscope are shown in Fig. 1.

A fine tungsten "point" similar in shape to the tip of a needle is attached to a tungsten loop whose temperature can be varied. When a positive potential of 6000 to 9000 volts is applied to an anode, electric fields of 30 to 50 million volts per centimeter are produced near the surface of the point. Such high fields "pull" electrons out of the metal even at room temperature. These electrons come out normal to the surface, travel in approximately straight lines in the field-free region of the tube, impinge on the fluorescent screen, and produce a visible pattern which can be observed by eye or photographed. If the densities of electron emission from various areas on the point emitter are non-uniform, the pattern on the screen shows corresponding variations in brightness. The geometric magnification is the point-to-screen distance divided by the radius of curvature of the point emitter. Magnifications of a million, with resolving powers of about 20 Angstroms, are readily attained. In special cases it may be possible to obtain magnifications of 10 million and resolving powers of three to five Angstroms.

In 1953, Becker [1] reported, "Recently Dr. Müller has given a number of lectures in which he has disclosed that protons can replace the electrons in producing an image on the fluorescent screen. In this case the potential of the point is positive and fields of about 200 million volts per centimeter are used. Hydrogen molecules are made to impinge on the point at room temperature and ions, which are presumably protons, are pulled off. In this case the resolving power is greater than in the electron case and considerably more structure can be seen." Extrapolating the above remarks, Mr. Fred L. Reynolds of the University of California Radiation Laboratory staff determined to investigate various "point" filament tubes to determine whether the point filament, hitherto negative, might be used as a source of positive ions by



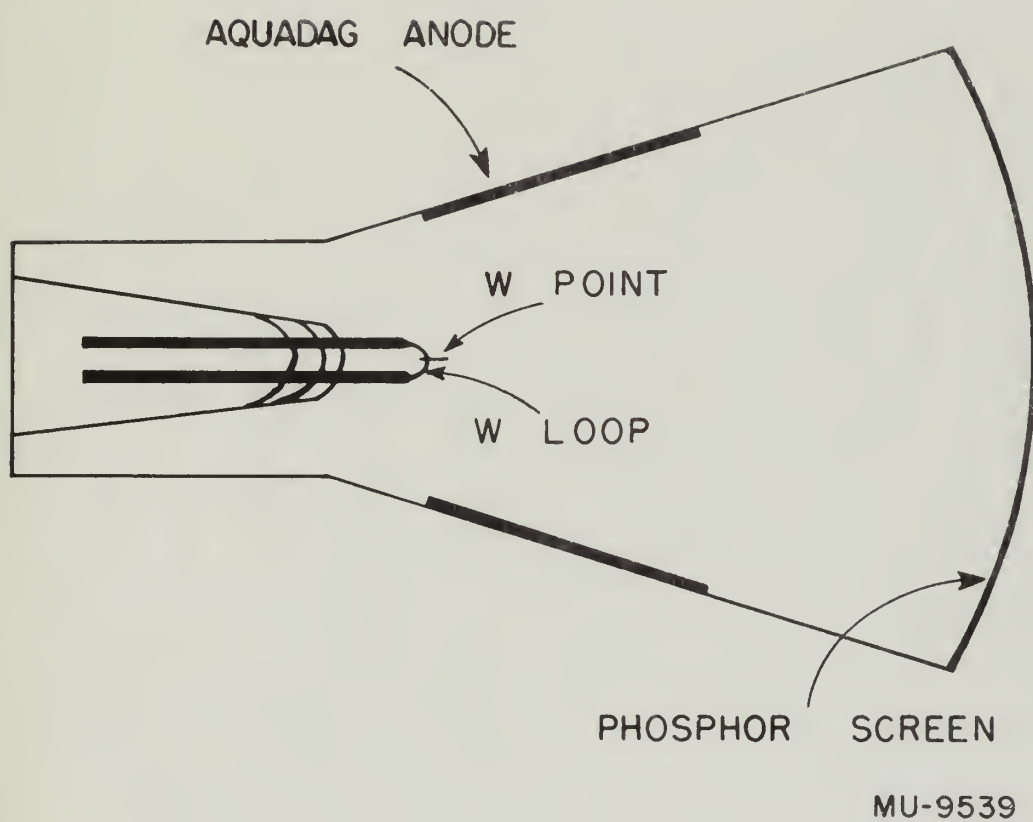


Fig. 1 A field emission microscope



reversing the field polarity, making the point positive, and impinging the sample on the point by suitable methods. He thought that since the first ionization potential of hydrogen (13.5 electron volts) is higher than most other elements, perhaps a point emitter that permits direct formation of positive ions of solid samples might prove an efficient ion source for mass spectrometer work, especially in mass abundance ratio work on the transuranic elements when samples are very small and hard to obtain.

The results of this project to date indicate that positive ions of any sample are formed by high fields at a point emitter. Solid samples can be ionized efficiently from such a point, but practical methods of accomplishing this are still in the process of evolution. I feel that although high field emission studies are in their infancy, continued research into this method of positive-ion formation by point emitters will greatly enhance the understanding of the fundamental processes of surface binding forces and ionization phenomena.



## CHAPTER II

### EMITTER FABRICATION

Needle-shaped tungsten field emitters have been mechanically ground, chemically etched, and electrolytically etched from wire stock; emission has been obtained even from the jagged end of a broken wire. Mechanically ground emitters were judged from their electrical behavior to have submicroscopic surface irregularities. Electron micrographs of the chemically etched emitters revealed undesirably complicated geometries. The electrolytic etch appears superior to the others because of the desirable electrical behavior of field emitters fabricated in this manner by Benjamin and Jenkins [2]. An electron micrograph of one of the emitters used in the work at University of California Radiation Laboratory is shown in Fig. 2. The radius of curvature of the point emitter at the tip is  $2.15 \times 10^{-5}$  centimeter, in excellent agreement with radii of curvature of about  $10^{-5}$  to  $10^{-4}$  centimeter reported by other workers.

The detailed fabrication procedure is as follows. The emitter blank, a short length of 20-mil tungsten wire (Callite 200H), which had been previously flashed in vacuum for 5 minutes at  $2800^{\circ}\text{C}$ , is spot-welded with a condenser discharge spot-welder to the apex of a support filament, which sometimes is beaded with uranium glass to ensure mechanical rigidity at the apex. The filament apex and emitter then are reduced to approximately 5-mil diameter and the entire assembly is smoothed, both operations by electrolytic etch. The emitter point is then shaped by a final electrolytic etch. For the several etches, an electrolyte of one-normal NaOH is used. The point emitter serves as one electrode. The second electrode is a 2-inch-diameter nickel helix submerged in the NaOH solution, which is held in a 100-milliliter glass beaker. The emitter blank is immersed periodically into the center axial region of the helix. A 60-cycle alternating potential of the order of 10 volts is satisfactory.

Continuous observation of the emitter tip by low-power microscope during the etch is recommended. The sharper points, which



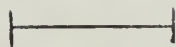
are a function of the cone angle of the point, usually occur immediately prior to fragmentation of the tip area by the etch. Additional observational checks by high-power microscope can confirm the relative condition of the point, but electron micrographs similar to Fig. 2 are required for accurate knowledge of the emitter point geometry.

Electron micrograph studies by Dyke [3] indicate that the emitter geometry is approximately paraboloidal. Surface irregularities are smoothed at the first test heating at  $2200^{\circ}\text{C}$  in vacuum, which permits surface migration of tungsten on tungsten.

Emitters produced by the techniques described have two geometric characteristics in common with a sphere mounted on an orthogonal cone: (a) the emitter closely approximates a sphere at polar angles of  $30^{\circ}$  or more relative to the apex, and (b) the shank is essentially conical at distances from the apex that are large compared with the tip radius. See Appendix I for field calculations.



1 MICRON



MU-9540

Fig. 2 Emitter point: electron micrograph

Figure 1



Figure 1

Figure 1: A dark, elongated, teardrop-shaped object, possibly a fossil or biological specimen, oriented vertically. Above the object is a horizontal scale bar with vertical end caps, labeled 'Figure 1'.

## CHAPTER III

### FABRICATION OF FLUORESCENT-SCREEN TUBES

Flat-faced and spherical tubes of varying sizes have been used. A standard spherical 500-milliliter pyrex flask may be utilized by adding a button-seal tungsten electrode at the equator of the sphere and a glass vacuum pump-out tube opposite the button seal.

First, electrical connection must be made to the interior of the pyrex envelope. This is accomplished as follows: Hanovia Liquid Bright Platinum No. 05 is brushed in a narrow band onto the interior of the envelope in an equatorial strip. After it is dry the tube is baked in air at 550° to 600°C for a half hour. The platinum salts are reduced to metallic platinum, which is fired into the glass. It may take as many as three coats to obtain a film that is opaque to visible light. Experience has been that if aluminum is evaporated over the platinum (when aluminizing the screen) a reaction takes place during evacuation bake-outs which results in thinning the aluminum-covered platinum film to the point where it is quite transparent. This has resulted in tube failures. In an attempt to remedy this failure, the platinum has been coated once with Hanovia Liquid Bright Gold No. 261. The platinum is better than gold for the base coats because of its better bake-in characteristics. After the gold is fired in for 15 minutes at 500°C, the tungsten lead in the button seal is etched clean inside by the electrolytic method. Then Dupont No. 4760 silver paint is brushed into the button seal region to make contact between the platinum-gold band and the tungsten lead. The silver paint is fired at 500°C for 10 to 15 minutes in air. The tube is thoroughly washed first with a detergent such as Tide, then finished off with a cleanser such as Alconox. It is permitted to drain dry.

Next, phosphor is applied by the lacquer flow method. Sylvania No. 2281, a willemite phosphor  $[\text{Zn}_2\text{SiO}_4: \text{Mn}(3\%)]$ , is suitable. Twenty grams of phosphor and 40 cubic centimeters of amyl acetate are poured into a quart ball mill half filled with 3/4-inch flint pebbles. The mill turns at about 15 rpm and is permitted to run for about three

ANALYSIS OF A COMPOUND CONTAINING 1.000g

1.000g of the compound was placed in a 100 ml. flask and

the flask was sealed with a stopper. The flask was then

placed in a water bath at 100°C. for 24 hours. The

flask was then cooled and the stopper was removed.

The flask was then placed in a water bath at 100°C. for

24 hours. The flask was then cooled and the stopper was

removed. The flask was then placed in a water bath at

100°C. for 24 hours. The flask was then cooled and the

stopper was removed. The flask was then placed in a

water bath at 100°C. for 24 hours. The flask was then

cooled and the stopper was removed. The flask was then

placed in a water bath at 100°C. for 24 hours. The

flask was then cooled and the stopper was removed. The

flask was then placed in a water bath at 100°C. for

24 hours. The flask was then cooled and the stopper was

removed. The flask was then placed in a water bath at

100°C. for 24 hours. The flask was then cooled and the

stopper was removed. The flask was then placed in a

water bath at 100°C. for 24 hours. The flask was then

cooled and the stopper was removed. The flask was then

placed in a water bath at 100°C. for 24 hours. The

flask was then cooled and the stopper was removed. The

flask was then placed in a water bath at 100°C. for

24 hours. The flask was then cooled and the stopper was

removed. The flask was then placed in a water bath at

100°C. for 24 hours. The flask was then cooled and the

stopper was removed. The flask was then placed in a

water bath at 100°C. for 24 hours. The flask was then

cooled and the stopper was removed. The flask was then

placed in a water bath at 100°C. for 24 hours. The

flask was then cooled and the stopper was removed. The

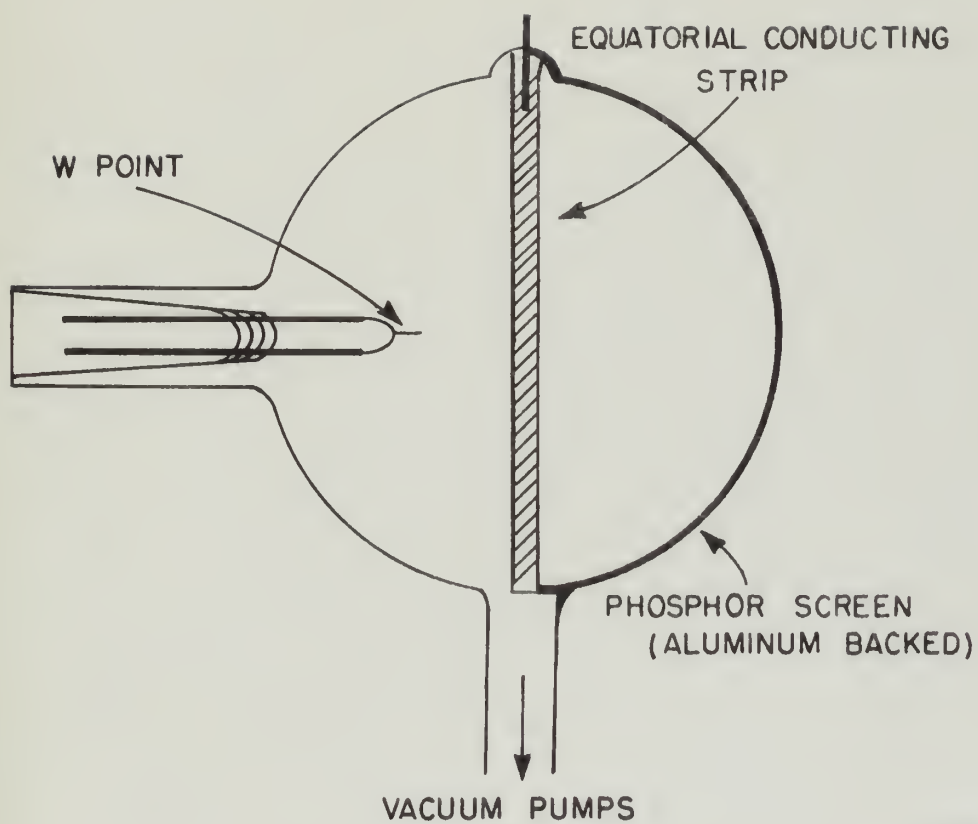
hours. The ball milling is an optional refinement required to break up aggregates. After milling, the suspension is poured into a container. An additional 10 cubic centimeters of amyl acetate is poured into the mill for rinsing purposes. The phosphor is allowed to settle, then 25 of the 50 cubic centimeters of amyl acetate is poured off. Then about 15 cubic centimeters Varnition V-21 (a label varnish) is added and thoroughly mixed.

Next the desired amount (gauged by experience) is poured into the tube. The tube is then immediately rotated about the axis normal to the center of the screen so that the lacquer spreads out uniformly over the surface. When the desired surface area has been covered with a very thin film, the tube is positioned upright with the wet phosphor concave downward. It is rotated moderately while a gentle stream of air is blown over the screen.

After the screen is dry, the tube is placed in an oven and given a 10-minute air bake at  $500^{\circ}\text{C}$  to remove the nitrocellulose lacquer. If necessary the tube may be carefully rinsed with distilled water to remove loose excess particles of phosphor.

Aluminizing then can be accomplished by the "metal carrier" technique, i. e. , using a thin nitrocellulose lacquer film on which the aluminum deposits. This procedure was reported by Ewald, [4] . A 6-turn spiral filament of tungsten (20-mil wire formed on a 6-32 bolt) is a good evaporating source. A tight-fitting slug of aluminum is placed axially on the inside of the spiral filament. The filament assembly is etched electrolytically in NaOH to remove oxide layers so as to prevent oxide fragments from evaporating as damaging splatterings to the screen. After the tube is aluminized the "metal carrier" is removed by baking in air at  $350^{\circ}\text{C}$  for a half hour. The phosphor tube envelope can now be joined to the point emitter as shown in completed form in Fig. 3.





MU-9541

Fig. 3 Fluorescent-screen tube

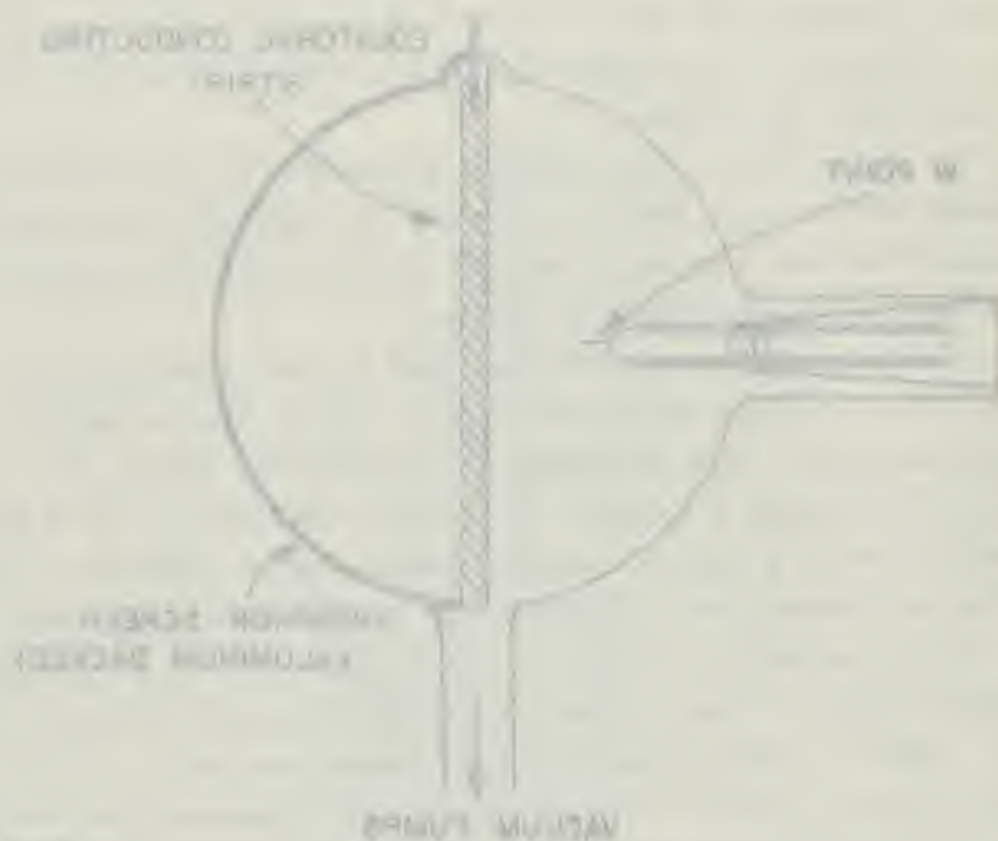


Fig. 1. Schematic diagram of the vacuum furnace.

## CHAPTER IV

### THE VACUUM SYSTEM

It is important to conduct point filament experiments in as good a vacuum as is practicable. This minimizes both the scattering of particles enroute--a limitation on resolution in phosphor pattern studies--and contamination of the point by residue atoms, which alters emission characteristics.

D. Alpert [5] has reported techniques for obtaining vacuum as low as  $10^{-10}$  millimeter of mercury in a straightforward and relatively simple manner without the use of special getters, charcoal traps, or refrigerants of any kind.

The innovation of the Alpert method is to utilize the pumping-out action of an ion gauge to evacuate the last few orders of magnitude of residual gas from a normally good vacuum ( $10^{-7}$  mm of mercury). This ion gauge pump-out is greatly facilitated if one uses a vacuum seal-off valve, metal throughout, which can be incorporated as a series part of the vacuum system.

The Alpert vacuum valve, modified slightly in the UCRL fabrication technique, permits the evacuation of manipulative systems as low as those previously attainable only in permanently sealed-off enclosures. It can withstand high-temperature bake-out and has proved to be of great value in ultra-high-vacuum work. Detailed construction data can be found in Alpert's report [5]. Leakage rates of the valve are extremely small, about  $10^{-10}$  liter per second. The valve can also serve as a crude needle valve, as is mentioned later under the experiment on ionization of hydrogen.

To assist in the necessary baking-out procedure, a double-walled, aluminum, reflector-type oven, glass-wool lined, 2 by 2 by 1.5 feet, was designed and constructed. This large-volume oven permitted complete bake-out of the important parts of the glass system at temperatures of about  $450^{\circ}\text{C}$ .

For convenience of operation the entire vacuum system was made portable by housing the mechanical forepump, oil diffusion pump,

# THE VARIOUS SYSTEMS

It is important to understand that the various systems are not all the same. The various systems have the same basic principle, but the details of the design are different. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems.

The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems.

The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems.

The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems.

The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems.

The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems. The various systems are designed to meet the needs of the various systems.

thermocouple indicator, two ion-gauge power supplies, and the glass system in a standard portable chassis rack, four feet high. The reflector oven base was attached to the chassis top and the "cake-pan" top of the oven was lowered onto the chassis whenever bake-out was desired. This arrangement furnished a compact portable vacuum system in which the oven bottom (chassis top) served as a convenient shoulder-high work platform. Tubes could be easily interchanged on this platform by using Alpert vacuum valves. The platform served as the necessary strength grip for the valves. The system is diagrammed in Fig. 4.



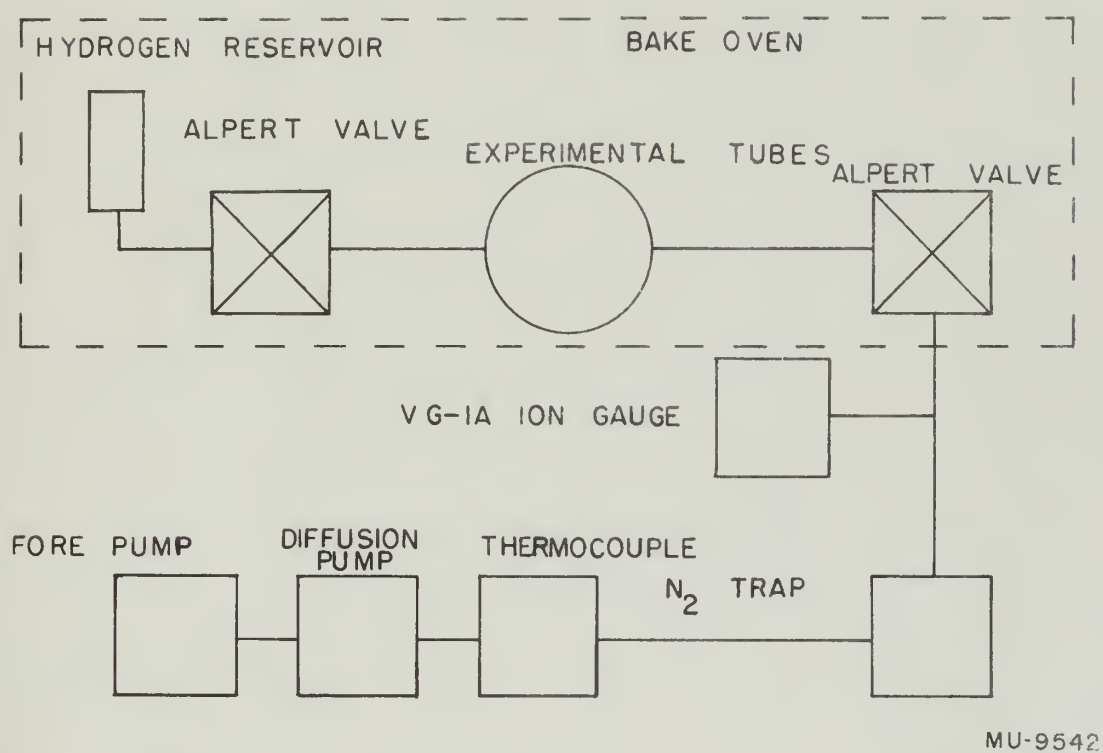


Fig. 4 The vacuum system



## CHAPTER V

### EXPERIMENTAL STUDIES

#### 1. Ionization of hydrogen.

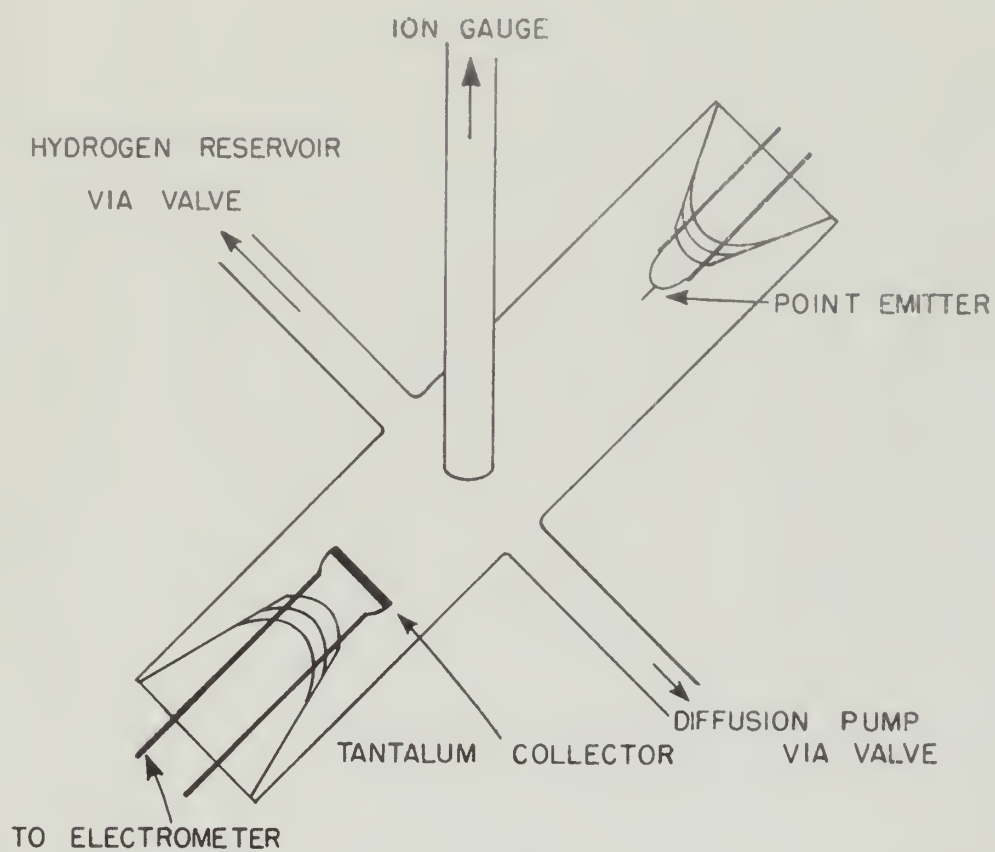
First, it was considered important to repeat Müller's experiment concerning proton emission to establish whether positive ions, and particularly protons, did indeed flow from the filament point.

A tantalum collector plate, approximately one square centimeter in area, was placed opposite an etched tungsten point filament at a distance of about thirteen centimeters in a cylindrical pyrex tube (Fig. 5) which had three tubular offshoots, respectively, for pure hydrogen input, ion gauge connection, and diffusion pump connection.

The concept of this run is that after bake-out, the tube is to be sealed off from the main system by an Alpert valve. An ion gauge then pumps down the tube to ultrahigh vacuum. With a Westinghouse (5966) ion gauge, pressures as low as  $1 \times 10^{-9}$  millimeter of mercury are reached. When a suitable high vacuum has been attained, pure hydrogen gas is introduced through a second Alpert valve used as a needle valve. The increasing hydrogen pressure is read on the 5966 ion gauge, which is running continuously during intake unless its upper pressure limit of approximately  $1 \times 10^{-4}$  millimeter of mercury is reached. When a desired pressure of hydrogen is reached, the tube is isolated by closing the hydrogen valve.

The hydrogen used is carefully prepared to minimize oxygen and water content. It is collected into a glass vial. The hydrogen vial is enclosed in a larger glass stub on the external side of an Alpert valve from the experimental tube. During bake-out and evacuation the Alpert valve is open to the system, ensuring evacuation of the stub holding the vial. When ultrahigh vacuum has been reached the Alpert valve input to the tube is closed and the hydrogen vial is shattered from the outside by the magnetic-striker technique. This creates a reservoir of pure hydrogen at almost atmospheric pressure which can be repeatedly used to introduce hydrogen into the experimental tube via the Alpert valve.





MU-9543

Fig. 5 Electrometer collection tube

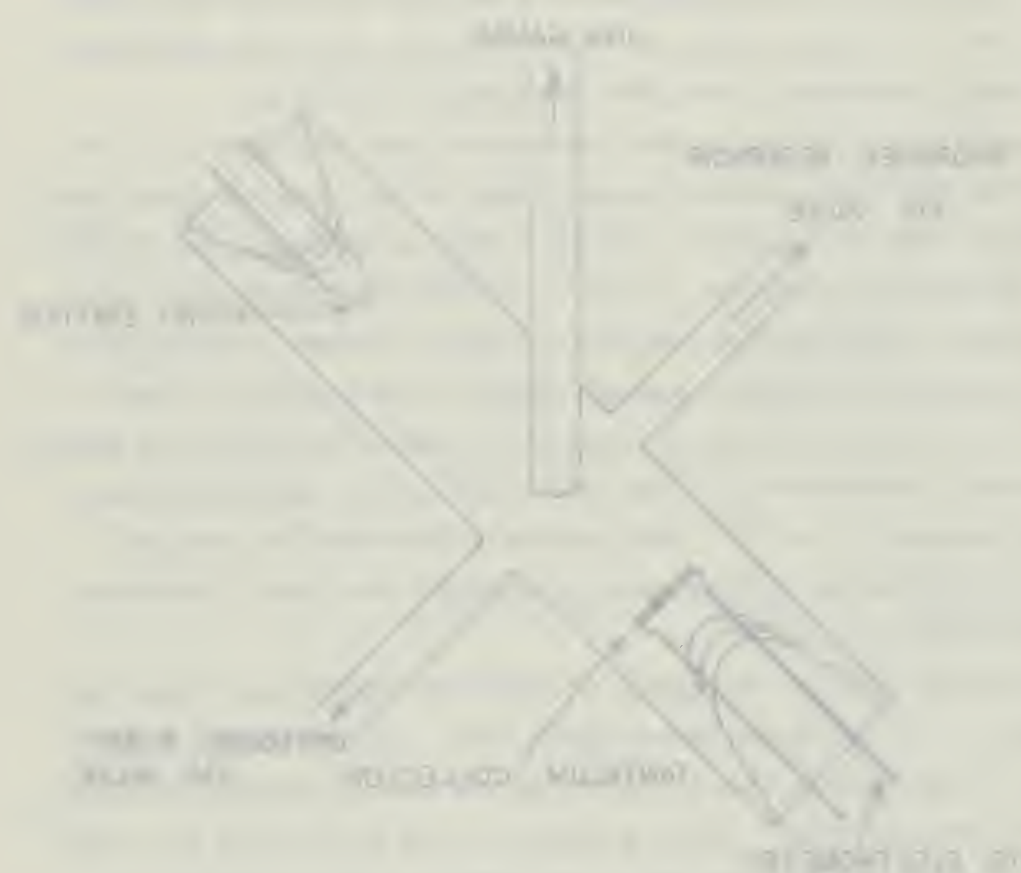
# Diagram of a Control System

Figure 1. Control System Diagram

The diagram illustrates a control system for a mechanical system. The system is composed of several interconnected components, including a motor, a controller, and a feedback loop. The motor is connected to the controller, which in turn controls the motor's operation. The feedback loop is used to monitor the system's performance and adjust the controller's output accordingly.

The diagram shows the following components and their connections:

- Motor:** The primary actuator in the system, connected to the controller.
- Controller:** The central processing unit that receives feedback and sends control signals to the motor.
- Feedback Loop:** A path that returns the system's output to the controller for comparison with the desired setpoint.



If one observes the ion gauge during the inlet period, pressures can be closely regulated.

The positive-ion current is measured on a positive-current-reading electrometer which measures currents as low as  $10^{-15}$  ampere.

In this experiment a regulated power supply of zero to 50 kilovolts was used. The point was positive and the tantalum collector plate was connected via the positive-current-reading electrometer to the grounded side of the high voltage supply. It was observed that if the 5966 ion gauge happened to be on while any voltage was applied to the point filament, an abnormally large electron current flowed via the point to the ion gauge electrodes. This occurred despite the lengthy, irregular, evacuated path of about 40 centimeters. This current overloaded the power supply. This current-overload phenomenon necessitated the addition of a Langmuir quartz-fiber mechanical pressure gauge on the experimental tube to record pressure readings whenever test voltages were applied.

After the experimental tube had been evacuated and sealed off as described earlier, hydrogen was introduced. Upon application of high voltage (37 kilovolts) with the point still at room temperature, positive ion current was observed to flow. There was a direct relation between pressure of hydrogen and positive current flow. This was evidenced by the continual decrease in positive-ion current as the hydrogen depleted itself by the pump-out action of hydrogen atoms sticking to the collector plate. Confirmation of this pressure-current relation was obtained by additional current observations as the pressure was deliberately reduced by evacuation through the diffusion pumps as regulated by the needle-valve action of an Alpert valve. In all cases the positive current decreased as the hydrogen pressure decreased.

Considering the initial purity of the vacuum and the care taken to ensure outgassing of both point filament and collector (by direct heating technique), it is to be concluded that hydrogen ions were formed and transferred by the high electric fields from the point to the collector plate, where they registered as positive ion current. Secondary electron emission at the collector plate was prevented by a suitable magnetic field.



Additional verification of this method of positive-ion formation has been recently reported in a letter by Marc Inghram [6] of the Argonne National Laboratory. This letter reports that a mass spectrometer was used as a detection device. It reports that 50 percent of the ions formed were  $H^+$  ions and the remaining ions were  $H_2^+$  ions.

## 2. Fluorescent screen patterns of point emitters.

The density of field-emission currents from a metal point is given by Becker [7] ,

$$j(\text{amp/cm}^2) = \frac{1.5 \times 10^{-6} F^2}{\phi} \approx 0.38 \phi^{3/2} \approx \frac{-6.6 \times 10^7 \phi^{3/2}}{F},$$

in which  $F$  is the field in volts/centimeter and  $\phi$  is the work function in electron volts. From this it follows that variations in  $j$  can be produced either by changes in  $F$  or in  $\phi$ :  $j$  will increase by a factor of 2 either if  $F$  is increased by about 5 percent or if  $\phi$  is decreased by about 3 percent. Hence contrasts in pattern intensity due to changes in  $F$  and  $\phi$  will usually be even greater than that in optical microscopy. If the surface of the point is very smooth near the tip,  $F$  will be uniform and variations in the pattern are to be ascribed to variations in  $\phi$ . A tungsten point which has been heated to  $2400^\circ K$  is sufficiently smooth so that variations in  $F$  are quite small for any angle less than  $20^\circ$  off the axis of the emitter. Variations in  $\phi$  are quite large, however, so that current densities from different planes of the single crystal may vary by factors of 1000. Since the tip of the point is so small, it is very likely that its hemispherical surface will expose all possible planes. Hence the patterns on a phosphor screen should show bright and dark regions which, from their symmetry, can be associated with definite crystallographic planes.

It is known that fluorescent-screen patterns can be used as detectors when solid materials depart from an emitter tip. Such departure of material greatly alters the emission characteristics of the point and hence indicates the conditions of temperature and voltage at which surface migrations and evaporation of solid materials as ions or neutral atoms occur.



Earlier experimenters have successfully used these variations of emission patterns to study the adsorption, migration, and evaporation characteristics of barium on a tungsten point filament. However, for mass spectrometer use a stable ion current is required to flow from the point anode. To achieve this one must ensure replenishment of the sample material at the tip of the emitter. In gaseous samples, as in the hydrogen experiment, this replenishment is brought about by kinetic impingement upon the point. For solid samples, however, some other mechanism must be used. If a preferential migration of solid sample particles to the point tip were to occur prior to vaporization conditions, it should be possible to obtain a stable ion current limited only by the migration rate. This ion current should be a function of surface temperature, sample density, and surface electric fields. This phase of the problem will assume greater importance as the art of using a point source develops. At present little information is available on migration rates of the heavy elements. Therefore emission-pattern studies form a separate, but interrelated, field to the point-source project.

To date the interest of the UCRL project in emission studies has been limited to developing techniques for emitter and tube fabrication discussed earlier. Fragmentary patterns of clean tungsten points similar to those described by Becker [7] were obtained. No precise analysis of crystal lattice structure has been attempted.

It is to be concluded from such fragmentary patterns and from examination of the reports of others in the field that emission-pattern studies do provide a tool to obtain further knowledge of the behavior of adsorbed heavy elements under high electric fields. A continuing increase in contribution to the literature on this subject can be expected. Two good reviews have been published by Jenkins [8] and Ashworth [9].

### 3. Transfer of solid samples as ions.

The decision was made to test directly whether solid samples, particularly the heavy elements, could be transferred as ions from a point emitter.

To facilitate detection of any ions transferred from the point, it was decided to use alpha-radioactive curium ( $\text{Cm}^{242}$ ) as the first sample



studied.

The concept of this experiment is that radioactive  $\text{Cm}^{242}$  is first deposited by careful mechanical contact of the emitter point with a sample-rich droplet. The sample is then dried onto the point by a heat lamp. A thin accelerator ring is placed in close proximity to the point (less than one centimeter). A removable and exchangeable collector plate is placed behind the accelerator ring. Numerous runs at different temperatures and voltages are made to determine if there is any temperature at which transfer of  $\text{Cm}^{242}$  is negligible in the absence of a field, but measurable upon application of an electric field.

The tube used for this experiment is diagrammed in Fig. 6. The use of a ball joint with its freedom of motion permits easy alignment of the point and the accelerator ring. The use of vacuum grease at these joints limited the workable vacuum to about  $10^{-7}$  millimeter of mercury.

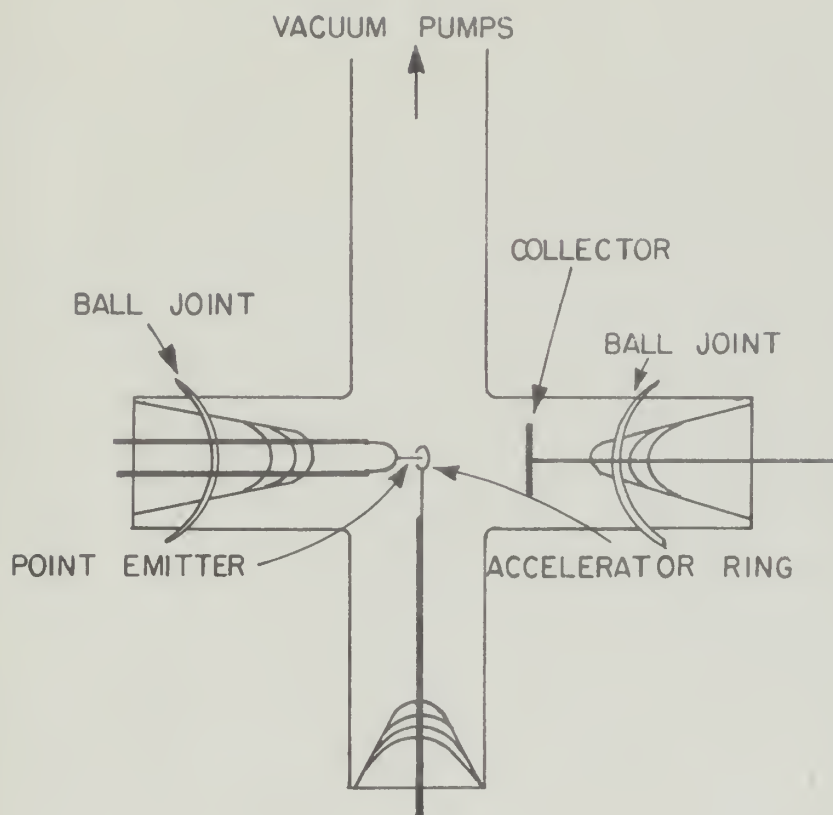
The solid sample was prepared in a totally enclosed work space, with rubber glove ports and glass windows, for health safety reasons. The original sample,  $\text{Cm}^{242}$  in hydrochloric acid, was evaporated down completely in an excess of nitric acid to ensure a conversion to the nitrate salt. The resultant salt was dissolved in very weak nitric acid and a droplet then was pipetted into a watch glass. The emitter point was carefully dipped into the droplet. An initial radioactive intensity of  $10^4$  counts per minute was placed in the immediate vicinity of the point. This was considered a safe and satisfactory activity level for the experiment. Temperatures of the filament were obtained by an optical pyrometer telescope.

The experimental data for 20 runs are listed in Table I. Background for all runs was less than 1 count per minute. All runs were for 30-minute duration, in sequence.

It is seen that there is no noticeable transfer of  $\text{Cm}^{242}$  below  $1500^\circ\text{K}$ , with or without voltage. This is interpreted to mean that none of the fields used were intense enough to pull off  $\text{Cm}^{242}$  from its surface bonding, even at  $1500^\circ\text{K}$ .

At  $1600^\circ\text{K}$ , a little  $\text{Cm}^{242}$  is transferred thermally (6 counts per minute). This temperature is therefore interpreted from this work to be the temperature at which higher-energy  $\text{Cm}^{242}$  atoms begin to





MU-9544

Fig. 6 Solid sample transfer tube



Table I

Ion-Transfer Studies ( $\text{Cm}^{242}$ )

Run No.	Temperature ( $^{\circ}$ Kelvin)	Potential (point to accelerator) (kilovolts)	cpm on collector plate
(1)	1100	0	<1
(2)	"	4	3
(3)	"	5	2
(4)	"	6	1.5
(5)	"	9	2
(6)	"	10	4
(7)	1300	0	<1
(8)	"	8	2
(9)	"	10	2
(10)	1500	0	<1
(11)	"	5	2
(12)	"	6	2.4
(13)	"	10	3
(14)	1600	0	6
(15)	"	2	8
(16)	"	3	54
(17)	"	10	55
(18)	"	12.5	30
(19)	"	0	6
(20)	"	10	29



vaporize thermally from a point emitter. At  $1600^{\circ}\text{K}$ , a noticeable amount of  $\text{Cm}^{242}$  is transferred when voltage is applied to the point (emitter positive, accelerator negative). This increase of transfer is a factor of 5 to 10 times thermal action for potentials over 3 kilovolts.

My conclusion is that  $\text{Cm}^{242}$  ions can be pulled from a point emitter by high electric fields after reaching a minimum temperature of approximately  $1600^{\circ}\text{K}$ .



## CHAPTER VI

### DISCUSSION OF RESULTS

The formation and "pulling-off" of hydrogen ions from a point emitter by high electric fields has been verified. The transfer of  $\text{Cm}^{242}$  from a point emitter has been indicated as occurring under certain conditions of temperature and field strength. Much work is yet to be done to make the point emitter a practical positive-ion source for mass spectrometry. In particular, the problem of supplying the sample continuously to the point is one demanding more knowledge of adsorption, migration, and vaporization characteristics of the heavy metals. Field-emission pattern studies may provide such information.

There is an indication in the  $\text{Cm}^{242}$  transfer runs that a migration of the sample to the point does occur. This is deduced from the apparent dropping off of  $\text{Cm}^{242}$  transferred, which in time levels off at approximately 30 counts per minute despite increased voltages. This strongly suggests that the positive ion emission from the point is limited to the  $\text{Cm}^{242}$  which arrives at the point by migration from regions of richer sample density. It is reasonable to assume that further investigation of migration rates will discover the optimum temperature-electric field relations to ensure high migration and hence practical ion currents, thereby permitting smaller samples than now necessary to be utilized.

In summation, it is my considered opinion that the point emitter will evolve as an extremely useful tool in the study of the fundamental physics of surface ionization. Whether or not it develops into a positive ion source for mass spectroscopy depends upon the future answering of many problems, notably sample replenishment.

So stands the project to date.



## BIBLIOGRAPHY

1. Becker, J. A. and Hartman, C. D. BELL TELEPHONE SYSTEM MONOGRAPH 2073 (1953)
2. Benjamin, M. and Jenkins, R. O. Proc. Roy. Soc. (London) (A) 176, 262 (1940)
3. Dyke, W. P. et al. Jour. Applied Physics, Vol. 24, No. 5, 570-576, May, 1953
4. Ewald, H. ELECTRONICS, 76-79, February, 1950
5. Alpert, D. Westinghouse Scientific Paper No. 1695, 1952
6. Inghram M., and Gomer, R. Jour. of Chem. Physics, 22, 1279, 1954
7. Becker, J. A. BELL SYSTEM TECH., JOUR. Vol. 30, 907, 1951
8. Jenkins, R. O. Reports on Progress in Physics, Vol. 9, 177, 1942-1943
9. Ashworth, F. Advances in Electronics, Vol. 3, 1, 1951

1	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
2	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
3	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
4	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
5	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
6	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
7	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
8	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
9	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males
10	Barrett, J. A. and Gardner, J. C.	Small Testosterone Production in Males

## APPENDIX I

### ELECTRIC POTENTIAL AND FIELD CALCULATIONS

(After Dyke et al. [3] )

For field calculations, the emitter and accelerating electrode surfaces can be thought of as different, but related, equipotential surfaces of a family of curves arising from theoretical consideration of a conducting sphere-on-orthogonal-cone, the "core", as shown in Fig. 7.

The potential distribution surrounding a charged, isolated, conducting sphere-on-orthogonal-cone is

$$V = \left[ V_R / R^n \right] \left[ r^n - a^{2n+1} r^{-n-1} \right] P_n (\cos \theta), \quad \text{Eq. (1)}$$

where  $r$  and  $\theta$  are ordinary plane polar coordinates referred to the center of the sphere as origin and to the pole of the sphere opposite its contact with the cone as zero direction;  $R$  is the actual electrode-separation distance,  $a$  is the theoretically derived radius of a sphere which gives rise to an equipotential that fits the true emitter geometry, and  $P_n$  is the Legendre function with  $n$  chosen so that the cone vanishes for  $\theta = \alpha$ , the exterior half angle of the cone. These quantities are shown in Fig. 7.

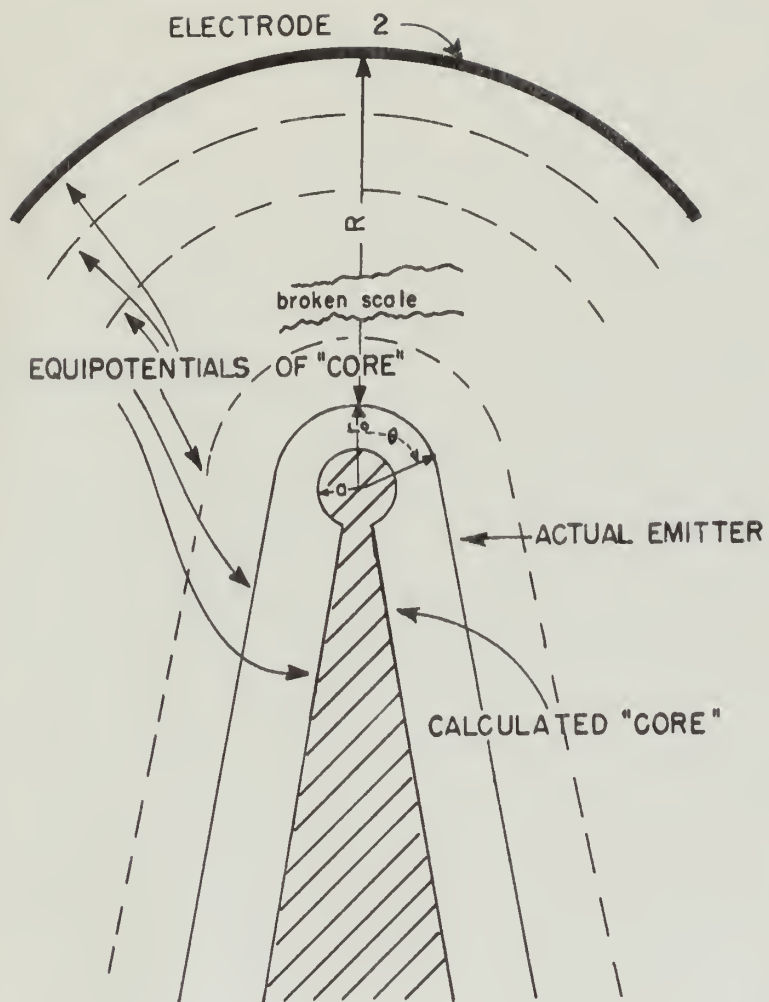
The theoretical collector electrode corresponding to the potential system defined by Eq. (1) can be determined by setting  $V = V_R$ . In this case the second term of the parentheses becomes negligible, and the polar equation of the anode is

$$r^n = R^n / P_n (\cos \theta). \quad \text{Eq. (2)}$$

The surface described by Eq. (2) somewhat resembles a paraboloid. For satisfactorily accurate field considerations, it can be readily approximated by construction of an experimental collector electrode.

Study of the characteristics of such a family of surfaces [Eq. (1)] reveals three important properties: (a) the neck constriction decreases





MU-9545

Fig. 7 Field calculation diagram



244-10

nozzle attached to nozzle 7 1/2

and disappears as the potential above that of the emitter increases (decreases for positive-ion work); (b) each surface approximates a sphere near the vertex; however, at intermediate distances from the vertex it is approximately conical with larger interior angle than that of the cone; (c) in the limit at very large distances from the apex the surface is parallel to the cone.

To obtain a desired equipotential approximating a given emitter and thus establish  $n$ ,  $a$ , and  $V$ , one has the three variables to work with in Eq. (1), i. e.,  $n$ , related through the Legendre function to the cone angle of the core,  $a$ , the radius of the theoretical core; and  $V$ , the resultant potential above that of the core. After some experimentation it is possible to find a combination of these parameters that leads to a surface closely approximating the dimensions of an emitter obtained by electron microscope pictures of that emitter.

For example, typical values for an emitter referred to in Fig. 7 and derived from Eq. (1) are  $n = 0.10$ ,  $a = 1.235 \times 10^{-5}$  centimeter, and  $r_o = 4.00 \times 10^{-5}$  centimeter ( $r_o$  is the value of  $r$  at  $\theta = 0$  on the surface that approximates the emitter).

The electric field,  $F_o$ , is the electric field at the emitter apex, i. e., the gradient of  $V$  evaluated at  $r = r_o$ ;  $\theta = 0$ .

$$F_o = \frac{r_o^{n-1}}{R^n} \left[ n + (n+1) (a/r_o)^{2n+1} \right] V_R . \quad \text{Eq. (3)}$$

$F_o$  is approximately 40 million volts per centimeter for the typical values of parameters given above if  $V_R$  equals approximately  $10^4$  volts and  $R = 1$  centimeter.









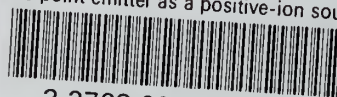


Thesis  
H52 Herron 28496  
Thesis  
H52 The point emitter as a  
positive-ion source.  
21 JUL 89 33579

Thesis  
H52 Herron 28496  
The point emitter as a  
positive-ion source.

thesH52

The point emitter as a positive-ion sour



3 2768 001 91901 2

DUDLEY KNOX LIBRARY